SCIENCE FOR CERAMIC PRODUCTION

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HEAT-INSULATING CORUNDUM CERAMICS BASED ON HOLLOW MICROSPHERES

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A high-temperature heat-insulating porous corundum ceramics is developed based on hollow microspheres produced using the plasma method. The ceramic properties, phase composition, and microstructure of the obtained material are studied.

Contemporary technologies make use of efficient heat-insulating fibrous materials, including silica, quartz, and kaolin fibers and glass ceramic fibers containing Al_2O_3 and ZrO_2. All the above fibers recrystallize under the prolonged effect of high temperature. In the course of service the fibers age and are destroyed, which presents a threat to operating personnel and the ambient environment [1 – 4].

The purpose of the present investigation is the development of porous corundum materials based on hollow Al_2O_3 microspheres to be used as lightweight heat-insulating materials.

Instead of fibers, the initial material in this case consisted of hollow corundum microspheres obtained by plasma melting that contain large single crystals resistant to recrystallization. Prolonged exposure to high temperatures does not cause migration of grain boundaries, and conditions for grain destruction can hardly be attained in practice [3].

The temperature dependence of the rate of crystal growth is known [3]:

$$D - D_0 = k\tau^{1/2},$$

where D is the grain final size at a certain temperature; D_0 is the initial grain size; k is a coefficient that grows exponentially with increasing temperature; τ is the heat-treatment duration.

In the case of use of microspheres, the difference $D - D_0$ is very small.

Materials comprised of hollow corundum microspheres have a low thermal conductivity, which makes them suitable for producing high-quality heat insulation [4].

However, microspheres of Al_2O_3 have low sintering activity, which is due to the fact that they are produced by plasma spraying of the oxide at high temperatures (over 2050°C). The resulting problem of sintering a material based on microspheres can be solved either by firing at extremely high temperatures or by binding the microspheres by special additives [5 – 7].

Such binding additives are often sol-gel compositions. A sol-gel binder covers the filler surface with a thin film and forms an ultradisperse phase that is active in sintering. An example of such a binder is hydrolyzed ethyl silicate modified with polyvinyl acetate dispersions [7], but such additives form eutectics with corundum, which decreases the classification temperature of the material (the classification temperature, or the heat resistance under load, is a special parameter of the behavior of a refractory material that is subjected to the combined effect of a load and an increased temperature and duration of treatment).

In the present study the additives facilitating sintering of microspheres were finely disperse corundum particles in the form of a slip used in the production of corundum ceramics at the Podol'skogneupor JSC and an alumina-based slip synthesized from industrially produced $Al(OH)_3$.

The additive was prepared by mixing Al_2O_3 powder, a polyvinyl alcohol solution, and water in a prescribed ratio. The mixture obtained was then mixed with the microspheres. The resulting mixture was placed in a mold and compacted by vibration. In vibration, the slip was absorbed by concave surfaces at contact sites of the microspheres. The molded articles were dried and fired in a furnace with lanthanum chromite heaters at temperatures of 1630°C (for composition 1 with the Podol'skogneupor binder) and 1550°C (for compo-

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TABLE 1

Sample	Apparent density, g/cm^3	Porosity, %	Compressive strength, MPa	Gas permeability, μm^2	Thermal conductivity at 1000°C, W/(m · K)	Classification temperature, °C
1	0.48	88.0	2.09	0.624	0.23	1360
2	0.38	90.5	1.16	0.505	0.41	1335

sition 2 with the binder synthesized from $Al(OH)_3$) with a hold of 1 and 3 h, respectively.

It should be noted that the drying conditions have a decisive effect on the finished-material quality. As a consequence of experiments, an optimum drying regime was selected: first, 24 h at a temperature of 80°C, and then 48 h at 120°C. This procedure provided for production of high-quality defect-free samples.

The porosity of the material was determined by filling the pore space with a liquid; the gas permeability was determined according to the method of GOST 11573–65 for porous refractories; the thermal conductivity was determined according to GOST 121-70–85 [8].

It was found that the thermal conductivity of a material based on corundum microspheres containing 0.5% binder at amounted to 0.28 W/(m · K) a temperature of 1000°C, and with 1.0%, 1.5%, 2%, and 2.5% binder it was 0.31, 0.39, 0.39, and 0.40 W/(m · K), respectively.

It should be noted that the thermal conductivity of both compositions was below the thermal conductivity of currently developed high-temperature fibrous heat-insulating materials, in particular, ones produced by Johns-Manville, a leading British company.

The classification temperature was determined in accordance with the international standard ISO 1893 : 1989. The classification temperature was taken to be equal to the temperature corresponding to 0.5% compressive deformation of the sample under a load of 0.05 N/mm^2. The experimental data are presented in Table 1.

The classification temperature of ceramic composition 1 was found to be 1360°C, the temperature of the start of deformation was 1250°C, and for ceramic composition 2 these quantities were 1335 and 1100°C, respectively.

The research data indicated that the samples of composition 2 have a lower apparent density and compressive strength and, accordingly, a greater porosity. The classification temperature of ceramic 1 is slightly higher than that of ceramic 2, which is related to the lower mechanical strength of the latter.

The microstructure was analyzed using a JEOL-JSM-T330A scanning electron microscope. It was found that the structure of the considered material is virtually equivalent to the model structure consisting of perfect spheres.

The production technology of this material is fairly simple and environmentally safe and allows an accelerated firing procedure, which contributes to saving of electricity.

The developed material can be widely used for lightweight heat insulation in high-temperature furnaces.

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